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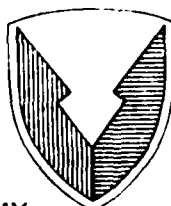
CORROSION BEHAVIOR OF HIGH DENSITY TUNGSTEN ALLOYS

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METALS RESEARCH DIVISION

August 1987



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ABSTRACT

Sodium Chloride

The corrosion behavior of high density tungsten alloys ($W \geq 90$ w/o) has been examined by electrochemical potentiostatic polarization methods and weight loss measurements in full immersion tests. Electrochemical tests were carried out in aqueous solutions, buffered to pH 4, 9, 12 with and without 0.1M NaCl and immersion tests in 5% NaCl solution. Pure tungsten and all the alloys studied undergo active-passive transitions and corrosion rates increase with increasing pH. The chloride ion generally increases corrosion rates slightly, but in pH12 solution, the chloride ion inhibits the anodic reaction to some extent. A 97W-2Ni-0.7Fe-0.3Co alloy was found to be the most corrosion resistant in solutions containing chloride ions. The data also suggests that copper as an alloying element accelerates corrosion of tungsten alloys in NaCl solution. Both general dissolution and localized attack at grain boundaries were observed. *Keywords:*



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INTRODUCTION

Sintered tungsten alloys are candidate materials for certain U.S. Army ammunition components because of their high density (17.0 to 19.0 g/cm³). The Army has a 10 year life requirement for ammunition stored in an uncontrolled environment. If the environment can be controlled, the storage life requirement can be extended to 20 years. Until recently, the corrosion of tungsten alloys has not been considered a problem. The disclosure by Andrew et al.¹ that a 90%W, 7.5%Ni, ~~2.5%Co~~ alloy readily corrodes when exposed to air saturated water vapor has caused the U.S. Army to reassess the situation. The work reported herein was aimed at evaluating the corrosion behavior of various sintered tungsten alloys under development by potentiostatic polarization methods in aqueous chloride solutions at varying pH's and by weight loss measurements after total immersion in chloride solution. The effects of controlled temperatures and humidity are also being assessed, but testing is still in progress.

EXPERIMENTAL PROCEDURE

The sintered tungsten alloys were fabricated using powder metallurgy techniques (isostatic compaction of the mixed metal powders followed by sintering in hydrogen).

The basic tungsten content² varied between 90 and 97%. The matrix compositions also varied and included elements such as Fe, Ni, Co, and Cu. Figure 1 is a micrograph of a typical liquid-phase sintered tungsten alloy microstructure showing rounded tungsten particles surrounded by a layer of matrix solid solution. In general, tungsten particles in these alloys were in contact and the matrix phase was confined to discontinuous or continuous interparticle interstices. The observed particle sizes varied from a few microns up to 80 μ , while the average mean particle size ranged from 25 to 40 μ , depending on the matrix composition and the sintering conditions. The matrix mean-free path was less than 2 μ and the tungsten grain contiguity was always from 2 to 3 particles. The alloy designations and compositions of the test materials are listed in Table 1. Pure tungsten was included for base line data. Some of the compositions were fabricated in-house; the remaining were supplied by a commercial vendor.

Table 1. COMPOSITIONS OF ALLOYS TESTED

Alloy	Composition, Wt. %				
	W	Ni	Fe	Cu	Co
Pure W	99.9				
8-11	90.1	1.6	1.1	0.2	
W ₁	90.1	1.6	0.5	0.5	0.1
27	90.0	1.5	1.0	0.4	0.1
31	90.0	1.5	0.5		0.1
33	90.0	1.5	0.5		
42	90.0	1.5	1.2		1.2
45	90.0	1.5	1.5		1.5
46	90.0	1.5	1.0		1.0

1. ANDREW, J. E., BAKIR, M. I., and HIRON, H. I. *Corrosion and Protection of Sintered Tungsten Alloy Ammunition Components*. Proc. of the Second Charlottesville Conference on High Density KI Penetrator Materials, October 1980, Sponsored by the Army Materials and Mechanics Research Center, Watertown, Massachusetts, in press.
2. CHIAO, W. F., and LARSON, J. R. *Matrix Chemistry and Mechanical Properties of High Density Tungsten Alloys*. Proc. of the Second Charlottesville Conference on High Density KI Penetrator Materials, October 1980, Sponsored by the Army Materials and Mechanics Research Center, Watertown, Massachusetts, in press.

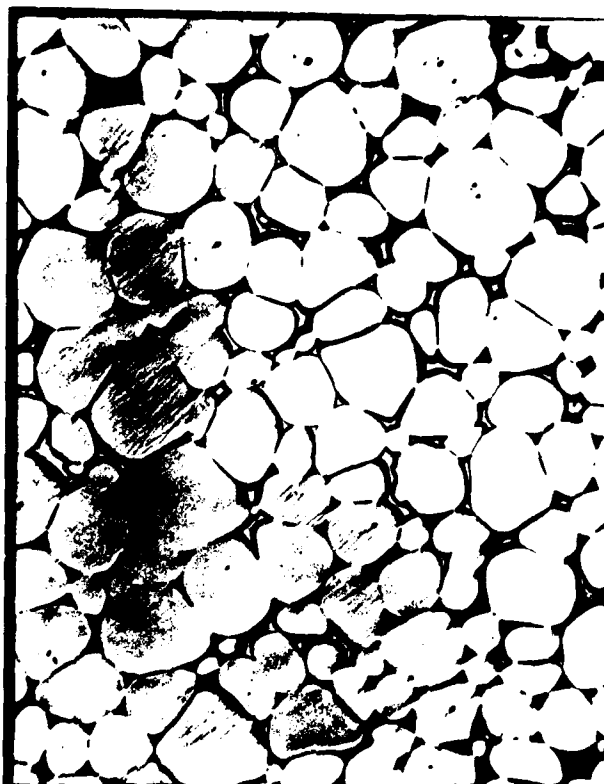


Figure 1. Typical microstructure of a high density tungsten alloy - unetched at 130X.

Specimens for both polarization and immersion tests were 0.250-inch in diameter and 0.500-inch in length. The specimens were polished with 240 and 600 Å silicon carbide paper, rinsed with distilled water, acetone degreased, air dried, and immediately used.

The solutions used for polarization tests were argon saturated and buffered to pH = 4, 9, and 12, with and without 0.1M NaCl. They were prepared from laboratory reagent chemicals and distilled water. The buffers used to fix the pH's included citric acid, disodium phosphate, and sodium hydrogen carbonate. Polarization measurements were also made on specimens in distilled water containing only the buffers to assess the effect of chloride ion. A 5% NaCl solution was used for the total immersion tests.

Polarization measurements were made utilizing the potential sweep method of potentiostatic polarization. The electrode potential was continuously changed at a constant rate of 4 V/hr, and current was simultaneously recorded. A Wenking potentiostat, in conjunction with a motor potentiometer for automatic programming of the operating potential and an X-Y recorder, was employed to automatically record current versus voltage. Prior to sweeping the potential, the specimen was allowed to remain in the solution for 90 minutes to let the potential come to a steady state corrosion potential before applying and varying the potential from the open circuit or corrosion potential to 2.0 V vs SCE. The polarization cell and general procedure have been described previously.³ In order to determine corrosion rates by

3. LEVY, M. *Corrosion* v. 23, no. 306, 1967.

extrapolation of Tafel slope to the corrosion potential, the anodic polarization curve was rerun between the corrosion potential and +0.2 V (more noble) at a slower scanning rate of 0.1 mV/sec.

One hundred cc of 5.0% NaCl solution was used to completely immerse test specimens for weight loss experiments. The volume of solution was appropriate for the surface area of specimen exposed. Immersion test specimens were weighed before and after 10 weeks of exposure. After testing and prior to weighing, exposed specimens were rinsed under running tap water, ultrasonically cleaned in distilled water for 10 minutes, rinsed in acetone, and air dried.

RESULTS AND DISCUSSION

Anodic Polarization

Pure Tungsten

Figure 2 shows the effect of pH and chloride ion on the anodic behavior of pure tungsten in buffered distilled water solutions. Tungsten undergoes an active/passive transition in all the solutions shown. Generally, the corrosion potential becomes more active, and the maximum dissolution current density and passive current density increase as the pH increases from 4 to 9 to 12. The greatest change occurs as the pH is increased from 4 to 9. Regardless of pH, the chloride ion produces a relatively slight increase in the magnitude of the maximum dissolution current. A distinct "knee" in the polarization curves is not observed in solutions of pH 4, with or without chloride ions; at the higher pH's, however, particularly in the solutions containing chloride ions, there are two knees attributed to the tungsten. None of the polarization curves show a transpassive region.

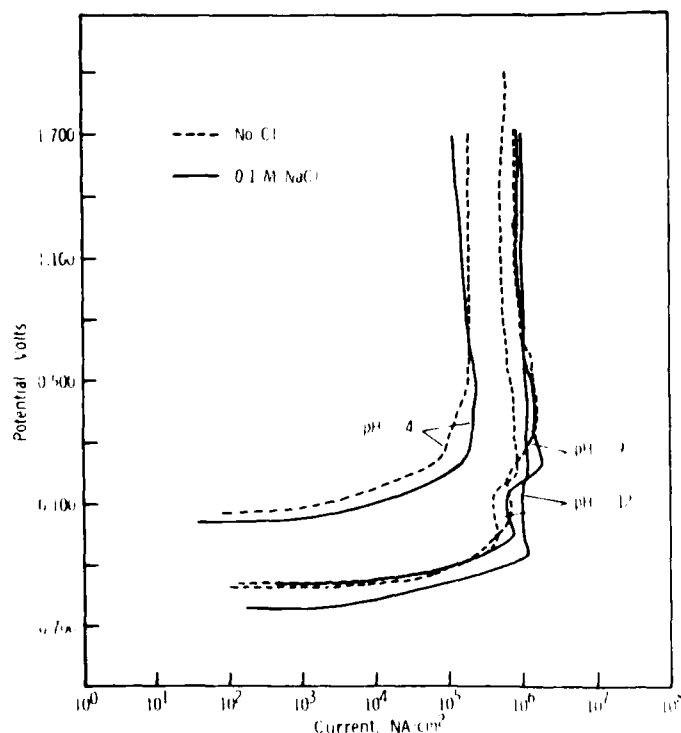


Figure 2. Polarization curves of W in various pH solutions with and without 0.1M NaCl.

Alloy W2 (97W, 1.6Ni, 0.7Fe, 0.5Cu, 0.1Co)

The effect of pH and chloride ion on the polarization curves for tungsten alloy W2 in buffered distilled water solutions is shown in Figure 3. The alloy undergoes an active/passive transition in all solutions. In all cases, the corrosion potential becomes more active as the pH is increased. In the absence of chloride ions, the maximum dissolution current density and the passive current density increase as pH increases from 4 to 12. Transpassivity occurs between 1.2 and 1.4 V vs SCE. The addition of chloride ions increases the dissolution current density significantly at potentials more noble than the corrosion potential. Above 0.500 V vs SCE, the polarization curves become essentially the same regardless of pH, and there is no transpassive region. At pH 9 and 12, they show three knees. The knee at ~0.500 V vs SCE is due to another phase introduced by the alloying elements. Below this potential, the two knees are attributed to tungsten. Comparing the anodic behavior of pure tungsten with that of alloy W2 in non-chloride solutions, the alloy is more easily passivated and exhibits a smaller passive current density. In the presence of chloride ions, the situation is reversed, and pure tungsten exhibits lower corrosion rates.

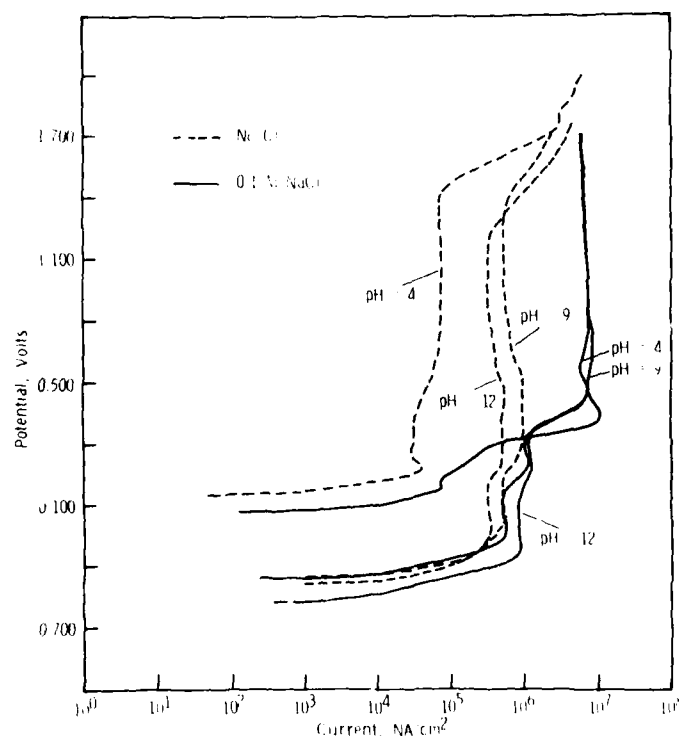


Figure 3. Polarization curves of 97W-1.6Ni-0.7Fe-0.5Cu-0.1Co in various pH solutions with and without 0.1M NaCl.

Alloy 31 (97W, 2Ni, 0.7Fe, 0.3Co)

Figure 4 shows the effect of pH and chloride ion on the polarization behavior of the alloy in buffered distilled water solutions. Active/passive transitions are observed for the alloy in all solutions shown. The corrosion potential becomes more active as pH is increased. The maximum dissolution current and passive current increase with increasing pH. A transpassive region is observed in all solutions (~1.4 V vs SCE). The chloride ions increased the dissolution rate of pure tungsten and the W2 alloy, but reduced the maximum dissolution rate and the passive current density of Alloy 31 in solutions of pH 9 and 12. The inhibition may be due to the

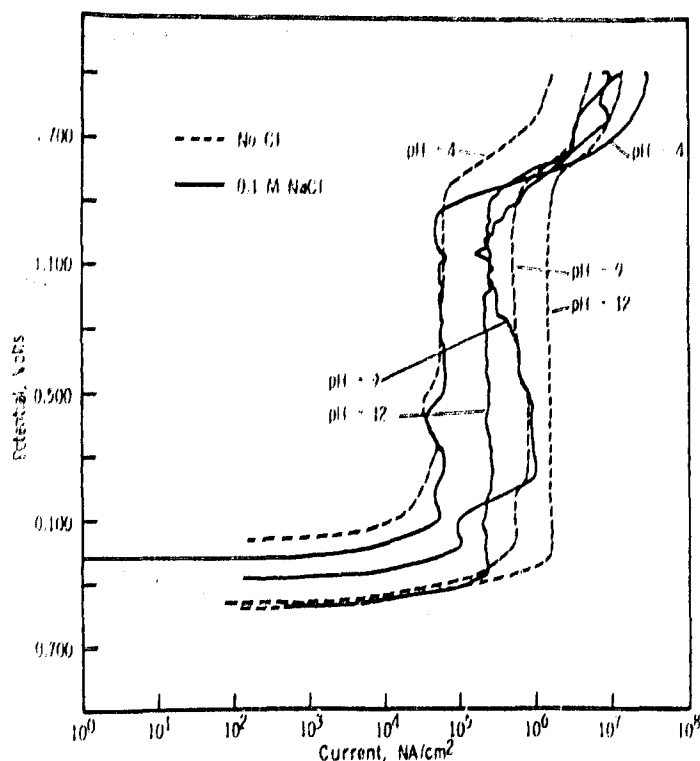


Figure 4. Polarization curves of 97W-2Ni-0.7Fe-0.3Co in various pH solutions with and without 0.1M NaCl.

formation of an insoluble protective tungsten-basic chloride film, or the chloride itself may stabilize the oxide film.^{3,4}

In the absence of chloride ions, the corrosion resistance of both alloys appears to be slightly superior to pure tungsten in a solution of pH4. There is little, if any, difference in the pH 9-12 range. When chloride ions are present (0.1M NaCl), alloy 31 exhibits better corrosion resistance (more noble corrosion potential, smaller maximum dissolution and passive currents) than the pure tungsten, but the W2 alloy appears to be less corrosion resistant (greater maximum dissolution current and passive current) than both pure tungsten and alloy 31. The major difference between the W2 and 31 compositions is the 0.5% Cu content of the W2 alloy. Thus, Cu may have a deleterious effect on corrosion resistance.

Other Alloys

Because of similarities in the anodic polarization behavior of the remaining alloys, additional polarization curves are not shown. The electrochemical parameters obtained for these alloys are contained in Tables 2-4. Generally, alloys containing copper are more susceptible to chloride ion attack.

Corrosion Rates by Tafel Slope Extrapolation

Tafel slopes were determined from slow scan polarization measurements and the corrosion current estimated by extrapolation to the corrosion potential. Corrosion rates in mpy calculated from the Faraday Equation are contained in Table 5. Pure

4. KOGER, J.W. Corrosion of Tungsten-3.5, Nickel-1.5 Iron and its Constituent Phases in Aqueous Chloride Solution - Preprint A-DA 6063, Union Carbide Oak Ridge Y-12 Plant, Oak Ridge, Tennessee, November 1974

Table 2. POLARIZATION CURVE PARAMETERS FOR ALLOYS IN pH4 SOLUTIONS

Materials	No NaCl					0.1M NaCl				
	ϕ_c	ϕ_m	I_m	I_p	ϕ_t	ϕ_c	ϕ_m	I_m	I_p	ϕ_t
Pure W	-0.155		0.15			-0.200	0.474	0.2	0.2	
97W-2.5Ni-0.5Fe	-0.250		0.03			-0.275	0.162	0.09	0.07	1.400
97W-2Ni-0.7Fe-0.3Co	-0.188	0.388	0.07	0.03	1.400	-0.250	0.175	0.08	0.03	1.200
96W-2Ni-1Fe-1Co	-0.250	0.350	0.07	0.02	1.310	-0.337	-0.063	0.30	0.07	1.300
95W-2.5Ni-1.25Fe-1.25Co	-0.231	0.387	0.06	0.04	1.320	-0.350	-0.100	0.20	0.08	1.325
90W-5Ni-2.5Fe-2.5Co	-0.252	0.262	0.10	0.06	1.025	-0.337	-0.025	10.0	0.1	1.400
97W-1.6Ni-1.1Fe-0.2Cu	-0.150	+0.263	0.09	0.08	N.D.	-0.200		9.0		0.023
97W-1.6Ni-1Fe-0.4Cu-0.1Co	-0.037	+0.137	0.06	0.02	1.400	-0.137	-0.052	1.0	0.03	1.400
97W-1.5Ni-0.7Fe-0.5Cu-0.1Co	-0.050	+0.050	0.03	0.02	1.400	-0.138	+0.329	10.0	8.0	

ϕ_c = Corrosion potential, volts

ϕ_m = Maximum dissolution current potential, volts

I_m = Maximum dissolution current density, mA/cm²

I_p = Passive current density, mA/cm²

ϕ_t = Transpassive potential; N.D. = Not Determined.

All Potentials refer to SCE.

Table 3. POLARIZATION CURVE PARAMETERS FOR ALLOYS IN pH9 SOLUTIONS

Materials	No NaCl					0.1M NaCl				
	ϕ_c	ϕ_m	I_m	I_p	ϕ_t	ϕ_c	ϕ_m	I_m	I_p	ϕ_t
Pure W	-0.400	0.562	1.05	0.9		-0.487	0.075	2.0	1.0	
97W-2.5Ni-0.5Fe	-0.487	0.275	1.00	0.7	1.250	-0.500	0.300	1.0	0.9	1.300
97W-2Ni-0.7Fe-0.3Co	-0.487	0.237	1.00	0.8	1.400	-0.375	0.300	1.05	0.15	1.100
96W-2Ni-1Fe-1Co	-0.475	0.350	1.00	0.8	1.150	-0.475	0.300	1.0	0.6	1.100
95W-2.5Ni-1.25Fe-1.25Co	-0.400	0.300	1.00	0.8	1.100	-0.500	0.300	1.0	0.8	1.200
90W-5Ni-2.5Fe-2.5Co	-0.512	0.250	1.00	0.8	1.200	-0.500	0.287	1.0	0.8	1.350
97W-1.6Ni-1Fe-0.2Cu	-0.400	0.425	1.00	0.8	N.D.	-0.400	0.074	1.2	1.0	0.000
97W-1.6Ni-1Fe-0.4Cu-0.1Co	-0.512	0.262	1.00	0.8	1.700	-0.364	0.650	3.0	2.0	1.600
97W-1.5Ni-0.7Fe-0.5Cu-0.1Co	-0.462	0.300	1.00	0.8	1.250	-0.462	0.450	9.0	8.0	

ϕ_c = Corrosion potential, volts

ϕ_m = Maximum dissolution current potential, volts

I_m = Maximum dissolution current density, mA/cm²

I_p = Passive current density, mA/cm²

ϕ_t = Transpassive potential; N.D. = Not Determined.

All potentials refer to SCE.

Table 4. POLARIZATION CURVE PARAMETERS FOR ALLOYS IN pH12 SOLUTIONS

Materials	No NaCl					0.1M NaCl				
	ϕ_c	ϕ_m	I_m	I_p	ϕ_t	ϕ_c	ϕ_m	I_m	I_p	ϕ_t
Pure W	-0.525	0.300	1.0	0.8		-0.612	0.340	1.05	1.0	
97W-2.5Ni-0.5Fe	-0.575	0.324	1.0	0.9	1.400	-0.640		1.00		1.200
97W-2Ni-0.7Fe-0.3Co	-0.525		1.5		1.500	-0.500		0.2		1.400
96W-2Ni-1Fe-1Co	-0.522	0.324	0.8	0.7	1.100	-0.550		1.00		N.D.
95W-2.5Ni-1.25Fe-1.25Co	-0.550		0.9		1.100	-0.562		1.00		N.D.
90W-5Ni-2.5Fe-2.5Co	-0.512	0.250	0.7	0.6	1.150	-0.575		1.00		1.300
97W-1.6Ni-1.1Fe-0.2Cu	-0.562		0.9		N.D.	-0.550	-0.375	0.2	0.15	0.300
97W-1.6Ni-1Fe-0.4Cu-0.1Co	-0.512		0.8		1.200	-0.431	0.520	4.00	1.0	1.400
97W-1.5Ni-0.7Fe-0.5Cu-0.1Co	-0.500	0.275	0.8	0.7	1.200	-0.580	0.450	9.00	8.0	

ϕ_c = Corrosion potential, volts

ϕ_m = Maximum dissolution current potential, volts

I_m = Maximum dissolution current density, mA/cm²

I_p = Passive current density, mA/cm²

ϕ_t = Transpassive potential; N.D. = Not Determined.

All potentials refer to SCE.

Table 5. CORROSION RATES OF ALLOYS IN AQUEOUS ENVIRONMENTS, MPY

Materials	No NaCl			0.1M NaCl		
	pH4	pH9	pH12	pH4	pH9	pH12
Pure W	0.02	0.04	1.2	0.02	0.09	0.17
27	0.23	0.47	2.5	0.02	0.04	0.58
31	0.03	0.03	1.2	0.01	0.10	0.02
35	0.02	0.07	1.8	0.01	0.05	0.09
42	0.04	0.07	1.4	0.03	0.08	0.11
45	0.27	0.11	0.72	0.17	0.17	0.21
46	0.02	0.02	1.1	0.02	0.05	0.02

tungsten and all the alloys tested exhibited acceptable initial corrosion rates (below 5 mpy). Pure tungsten appears to be more corrosion resistant than most of the alloys in aqueous solutions. Generally, corrosion rates increase with increasing pH, except in solutions of pH12 containing chloride ions that show an inhibiting effect. The copper containing alloy exhibited the highest corrosion rate in most of the solutions.

Full Immersion Tests in 5% NaCl Solution

It should be noted that a constant area of each specimen was in contact with the glass beaker containing the solution. Within a week of exposure, alloys not containing copper (alloys 31, 42, and 45) formed a rust-colored reaction product (WO_2) in the area of contact, indicative of crevice-type corrosion. As exposure continued (up to 10 weeks), the reaction product spread over the entire surface of the specimen. This type of reaction product was not observed on pure tungsten and alloys containing copper (alloys W2 and 27), but the alloys containing copper formed a yellow reaction product, identified as WO_3 . The pure tungsten appeared to be

Table 6. RESULTS OF FULL IMMERSION TESTS

Specimen Number	Wt. Loss* (g)	Corrosion Rate (mpy)	Observation		
			After Test Solution Appearance	Rust On Specimen Surface	Rust Deposited on Beaker Bottom
Pure W1	0.0006	0.11	Clear	None	None
Pure W2	0.0004	0.08			
W2-1	0.0240	4.63	Milky	None	Extensive, Yellow
W2-2	0.0249	4.82			
27-1	0.0183	3.47	Milky	Slight Red-Brown	Some, Yellow
31-1	0.0112	2.12	Clear	Extensive Red-Brown	Little Yellow, Red-Brown
31-2	0.0100	1.93			
42-1	0.0052	1.00			
42-2	0.0049	0.94			
45-1	0.0039	0.77			
45-3	0.0029	0.57			

*Specimen Surface Area: 90.5 in².

unaffected. The weight loss data contained in Table 6 show that pure tungsten is the most corrosion resistant, followed by alloys containing no Cu and alloys containing Cu in order of decreasing merit. The better performance of the non-Cu containing alloys is attributed to the formation of the more protective WO₂. The detrimental effect of Cu was shown in both electrochemical and immersion tests.

SEM examination of the corroded surface of the W2 alloy was made after 10 weeks of exposure. The micrographs in Figures 5a and 5b show that the matrix phase corrodes more readily than the primary tungsten grain phase and that tungsten grain pickout can occur due to this intergranular attack. Thus, both general dissolution and localized attack at the grain boundaries are operative.

CONCLUSIONS

Anodic polarization curves show that pure tungsten and all the alloys studied undergo an active-passive transition; also, as pH increases, the corrosion potential becomes more active and the anodic current increases. The chloride ion increases the anodic dissolution of tungsten and most of the alloys in pH4 and 9 solutions, but, in solutions of pH12, there is little or no effect.

Based on the magnitude of the maximum dissolution and passive currents, the 97W-2Ni-0.7Fe-0.3Co alloy is the most corrosion resistant material studied in solution of pH4. The chloride ion inhibits the anodic reaction of this alloy in solutions of pH9 and 12.

In the absence of chloride ion, the alloys appear to be more corrosion resistant than pure tungsten in solutions of pH4. Alloys containing copper appear to be the least corrosion resistant of the materials studied in 0.1M NaCl solution.

In general, corrosion rate data obtained by the Tafel slope extrapolation method support the aforementioned conclusions. Data obtained from total immersion tests in 5% NaCl solution indicate that pure tungsten has the lowest corrosion



(a)



(b)

Figure 5. SEM micrographs showing (a) rapid grain boundary corrosion (matrix), and (b) tungsten grain pickout after 10 weeks exposure of 97W-1.6Ni-0.7Fe-0.5Cu-0.1Co alloy in 5% NaCl solution (350X).

rate and that alloys containing copper have the highest corrosion rates. Regardless of the method of test, it appears that copper increases the corrosion rate of tungsten alloys in chloride solutions.

Based on corrosion rates (penetration in mpy) calculated from extrapolated Tafel slopes and weight loss measurements, all the materials studied exhibit satisfactory behavior in aqueous solutions (less than 10 mpy); but the SEM micrographs of the W2 alloy, after 10 weeks of immersion in 5% NaCl solution, showed that the matrix phase is attacked more readily than the tungsten grain, and penetration in those areas can exceed the calculated penetration rates. Further, this type of grain boundary attack can result in loss of strength and ductility.

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2 ATTN: SLCMT-IML

2 Authors

U.S. Army Materials Technology Laboratory, Watertown, Massachusetts 02172-0001 CORROSION BEHAVIOR OF HIGH DENSITY TUNGSTEN ALLOYS Milton Levy and Frank Chang	AD UNCLASSIFIED UNLIMITED DISTRIBUTION	Technical Report MTL TR 87-37, August 1987, 12 pp - illus-tables	Key Words Tungsten alloys Density Corrosion resistance	The corrosion behavior of high density tungsten alloys ($M > 90$ w/o) has been examined by electrochemical potentiostatic polarization methods and weight loss measurements in full immersion tests. Electrochemical tests were carried out in aqueous solutions, buffered to pH 4, 9, 12 with and without 0.1M NaCl and immersion tests in 5% NaCl solution. Pure tungsten and all the alloys studied undergo active-passive transitions and corrosion rates increase with increasing pH. The chloride ion generally increases corrosion rates slightly, but in pH12 solution, the chloride ion inhibits the anodic reaction to some extent. A 97W-2Ni-0.7Fe-0.3Co alloy was found to be the most corrosion resistant in solutions containing chloride ions. The data also suggest that copper as an alloying element accelerates corrosion of tungsten alloys in NaCl solution. Both general dissolution and localized attack at grain boundaries were observed.
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